Professional Association of Natural Rubber in Africa (ANRA)

PROJECT ON QUALITY IMPROVEMENT AND QUALITY CONTROL
OF AFRICAN NATURAL RUBBER
CFC SUBSIDY No.28

CIRAD

Report on the Statistics – Information Technology mission for the project on Quality Improvement and Quality Control of African Natural Rubber

Cameroon

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Mission schedule

This statistics-information technology mission proceeded as follows:

18 June	Arrival in Douala
19 June	Douala: Smallholder session – Results of the first set of proficiency tests
20 – 21 June	IRAD Ekona: Statistics talk – Tour of the Tiko factory
22 June	Douala: Statistics talk – Presentations by laboratory managers
23 June	Departure from Douala

SUMMARY

This mission to Cameroon in connection with the CFC project on the improvement of natural rubber quality was used to take stock of the first set of proficiency tests on a regional scale, which took place in November 1999. The criteria used to assess the performance of the laboratories were discussed. The broad outlines of the same protocol were adopted for the next three series of tests, which have been scheduled between now and the end of the project.

In addition, additional statistics applied to quality control and innovations in the JMP software were also presented to the central laboratory managers.

<u>**CONTENTS**</u>

INTRODUCTION

The CFC project for the quality improvement and quality control of African natural rubber chose to strengthen the capability of the central rubber analysis laboratories, by launching a system of proficiency tests in the region based on Standard African Rubber (SAR) methods. To that end, during an initial statistics mission in 1999, a protocol was defined for these tests and modern computing equipment was installed in each laboratory. An initial cycle of tests took place at the end of 1999.

This second mission in June 2001 was used to take stock of the first cycle. It was possible to clarify a certain number of points and start organizing subsequent cycles, which are due to take place between now and the end of the project.

In addition to the proficiency tests, it was useful for the laboratory managers to acquire a few statistical techniques applied to quality control and process management. They were presented during this mission.

I – OVERVIEW OF THE FIRST SET OF PROFICIENCY TESTS – ORGANIZATION OF SUBSEQUENT TESTS

First set of proficiency tests

The first set of tests took place in November 1999. The problems encountered by both the organizers and the participants did not appear to be insurmountable. It appeared that the time taken to prepare samples should not be underestimated, and could take several days. In addition, as dispatches by express mail were very expensive, it was decided that each CTL would go away from this workshop with the samples required for the following three sets of tests.

The results from this first set of tests were used to define the format of the results sheets. These sheets (annex 1) carry all the information enabling the manager to judge the ability of his laboratory to measure a given property.

The sheets have five sections:

- **Sorted results for all the laboratories taking part.** The values listed are the means of measurements taken on 5 samples analysed on the same apparatus. The manager can thus check that there have not been any errors in the processing of his data, and position himself in relation to the others. The mean and standard deviation are indicated for these series of results. The last row in this table is the median of the results obtained. By convention, it is this value that is taken as the bale value reference.

- **The Z-scores.** This is an essential result of the proficiency test for a given apparatus. It measures the apparatus calibration defect. A Z-score over 2 is judged to be unreliable, given the dispersal of results that is allowed. A Z-score over 3 indicates that there is a problem in the measuring chain. In all cases, laboratory managers must try to improve their Z-score from one cycle to the next (i.e. obtain the lowest possible value), by adjusting their equipment so as always to move nearer to the reference value.

- **Repeatability.** This is the standard deviation for measurements taken on the 5 samples analysed on the same apparatus. The mean of the set of repeatability standard deviations obtained is calculated.

- **The K-scores.** This score indicates whether the apparatus repeatability is better than (K-score under 1) or worse than (K-score over 1) the mean of the participants. A K-score over 2 indicates that there is high dispersal of the 5 measurements and it needs to be checked whether there is an uncontrolled source of random error.

- **Youden plot.** This graph presents the values obtained for one grade depending on the other. It makes it possible to see at a glance whether the equipment obtains systematically over- or underestimated results, or on the other hand whether it is within the mean of the other laboratories.

Clear differences were seen during this first cycle in the know-how of the different participants. It turns out that the results were often highly dispersed and it was sometimes difficult to determine which were wrong and which were right. Under these conditions of uncertainty, it was judged more reasonable to initially fix quite broad limits for defining normal variability of results. The target standard deviations, i.e. the inter-laboratory standard deviation values expected if the differences between laboratories only came from "normal" random variations, were therefore chosen quite broadly. The following table gives the values used.

PROPERTY	Target SD
Dirt	0.007
Ash	0.01
Volatile	0.03
N	0.04
P0	4
P30	4
PRI	3
MV	1.5
Colour	1

Result sheets for the first cycle were sent to all the participants. Depending on the scores obtained, the laboratory managers will judge whether they need to take corrective measures to bring their measurements into line with the reference value (i.e. the "true" value by convention).

It will not be possible to say whether the proficiency tests system introduced leads to any appreciable progress in reducing result dispersal until the second series of tests has been completed.

Organization of subsequent tests

The second set of tests is due to take place soon. The protocols for sample preparation and data processing are identical to those used for the first set of tests.

As there are plans for plantation company laboratories to take part in this cycle, modifications are required in the automatic data processing procedure (annex 2), so that it is more able to process trials involving a given number of participants. This modification will be made by the project statistician in Montpellier, and passed on to the project leader.

Other changes to this procedure to give it an even more general scope may be carried out by IT student trainees spending time in the laboratory. It may be that during their university course, the IT students from the University of Buea, for example, may acquire the necessary wherewithal to programme macros in Visual Basic under Excel.

II – ADDITIONAL STATISTICS APPLIED TO QUALITY CONTROL

Part of the mission was devoted to talks on applied statistics. One talk explained the different types of graphs in the summary report drafted by the project managers at the end of the first series (Box and whiskers plot, Quantile-Quantile Plot, mean comparison circles, etc.). Another talk described the innovations in the new version of the JMP software, with a demonstration copy. The innovations mainly consist of the possibility of writing scripts (sorts of programs) and paired comparison tests. The reading of data from other softwares and transfer of results to other softwares have been made easier, as has carrying out the same analysis on several groups of individuals. The price of this software for research centres is 2,700 French francs.

Using check charts in the factories, and their creation with JMP were also mentioned. Check charts are graphs showing how production varies over time. In fact, it is a sheet of graph paper on which results measured on samples taken periodically from output are recorded according to time. Lines corresponding to the theoretical mean value of the measurements and to the limits they must not exceed are plotted beforehand on the graph. These limit values are generally determined from a pilot experiment during which particular attention is paid to keeping the production process under control. The limits can be calculated and the charts can be plotted by JMP.

Lastly, a reminder was given of the laws of error composition. These laws are used to know the error affecting the result of a calculation based on known magnitudes with a given degree of error or uncertainty. For example, this is the case for PRI, which is calculated from the P0 and P30 values.

In the general case of a magnitude Y derived from n magnitudes X1,...,Xn by the formula Y=f(X1,...,Xn), a distinction needs to be made between two cases, depending on whether the errors affecting X1,...,Xn are systematic or random. If the errors are systematic (calibration defect) and amount to e1,...,en, the error affecting Y will be equal to $\sum_{i=1}^{n}\frac{\partial f}{\partial X_i}e_i$. If the errors are random (lack of repeatability) and have standard deviations s1,...,sn, then the standard deviation of the error affecting Y will be $\sqrt{\sum_{i=1}^{n}\left(\frac{\partial f}{\partial X_i}s_i\right)^2}$.

CONCLUSION

This first cycle of proficiency tests on a regional scale is a major step towards the constitution of a standard African rubber. Two new series of trials have already been scheduled between now and the end of the project. They will probably show convergence between the participating laboratories. In order to achieve sustainable agreement between the laboratories, it is to be hoped that this system will be able to find the necessary resources to ensure its operation in the coming years.

13/07/2001 **Annex 1**

ANRA/ACNA **SAR Proficiency Testing Scheme/Essais interlab. SAR**

Round #/Série n° 1 Lab #/Laboratoire n° 5

Property/Propriété : **P0**

All participants results (5 samples means)/Ensemble des résultats (Moy. 5 échant.) :

	Off Latex		10 CV
	38,100		34,500
	39,700		36,400
	40,200		37,100
Your/Votre Equipt. 1 ->	*41,600*	*Your/Votre Equipt. 2 ->*	*39,800*
Your/Votre Equipt. 2 ->	*43,000*	*Your/Votre Equipt. 1 ->*	*40,000*
	45,800		42,400
	51,200		47,800
	57,200		53,000
Grand mean/Moy. Gnle. :	44,600	Grand mean/Moy. Gnle. :	41,375
Betw. Lab STD:	6,557	Betw. Lab STD:	6,235
Reference (Median):	**42,300**	**Reference (Median):**	**39,900**

This Lab Z-Scores/Z-scores pour ce laboratoire :

Equipt.	Off Latex		10 CV
1	0,175		0,025
2	0,175		0,025

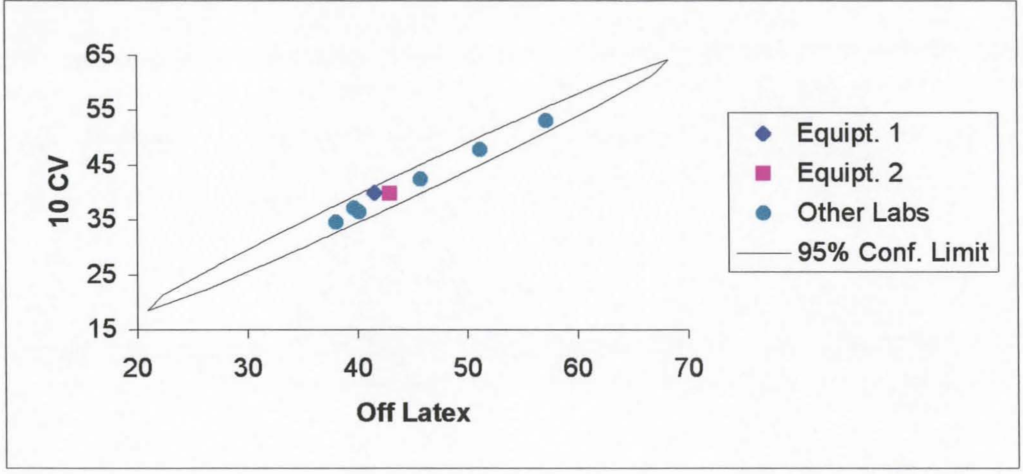
Repetability Analysis/Analyse de répétabilité :

	Off Latex		10 CV
Mean Repet/Répét. Moy. :	0,721		1,045
Repet. Equipt. 1	0,548		0,707
Repet. Equipt. 2	0,000		0,837

This lab K-Scores/K-Scores pour ce laboratoire :

K-Scores Equipt. 1	0,760		0,677
K-Scores Equipt. 2	0,000		0,801

Youden Plot/Ellipse de Youden :



Annex 2

Statistical analysis of SAR trials

Processing procedure (Draft)

Introduction

At the beginning of a test cycle, each laboratory receives a diskette with the Excel spreadsheet Excel "SARInputForm.xls", in which it records the results of its measurements for all its measuring lines.

The diskettes are returned to ANRA, which carries out analyses as soon as all the diskettes have been received.

In brief, the analysis procedure is as follows:

- Production under Excel of a common table for all the laboratories.

- Calculation of the Z and K scores for each set of measuring lines, for each property and each grade.

- Plotting of the Youden graph for each property.

- Printout of result sheets for each laboratory.

- Backup copy of the common table for the records.

This document details the IT and statistics aspects of the test cycle. This document is accompanied by a reference diskette containing two Excel files: "SARInputForm.xls" and "SARTogether.xls". This diskette should be kept.

SARInputform.xls is the blank input form and SARTogether.xls is the spreadsheet on which the results from the labs will be compiled and will be used for statistics calculations.

When the document and diskette are received, a directory, which will be called SARWork is to be created under the hard disk root on the ANRA computer, and the two files on the diskette will be copied to it. These two operations (directory creation and file copy) will be done with Windows Explorer. The files will be copied from the diskette whenever it is suspected that the two files on the hard disk are not identical to the reference files, for one reason or another (change of computer, hard disk crash, virus infection, accidental deletion or write-over)..

I) General

Before each cycle, when samples are being prepared at ANRA, the organizers prepare one diskette per laboratory (i.e. 5 diskettes in all). The diskettes used for the previous cycles can be re-used.

Preparation consists in:

- deleting anything already on the diskettes,

- copying a blank input form to each diskette, i.e. the "SARInputform.xls" file, which is in the "SARWork" directory on the hard disk, and that file only,

- sticking a label drawn up as follows, on each diskette (over previous labels if necessary):

ANRA SAR proficiency testing scheme **ACNA** Essais interlabos réseau SAR
Round No/Série n°: *<Numéro de la série>* Lab/Laboratoire: *<Nom du laboratoire>*
Insert diskette and open the "SARInputForm" Excel sheet to enter results ***Insérer la disquette et ouvrir la feuille Excel "SARInputForm" pour entrer les résultats***

The *Round No.* and *Lab name* (addressee of the diskette) must be filled in by ANRA.

The diskette will be sent with a covering letter drawn up roughly as follows:

- Your lab has *<n=1,2 or 3>* measuring lines and is therefore receiving *<n>* sets of samples numbered 1 to *<n>* for Latex grade and *<n>* sets of samples numbered 1 to *<n>* for grade 10 CV.

- Irrespective of grade, the samples in each set should be analysed on the measuring line bearing the same number as the set of samples (e.g. samples in set 1 are to be analysed on measuring line No. 1).

- The results should be entered in the Excel spreadsheet in the spaces defined by the following two principles:

. The results obtained for samples in set No X should be entered in the <u>columns</u> entitled "SAMPLE SET No. X", in the zone corresponding to their grade (Latex at the top, 10 CV lower down).

. They should be entered in the <u>rows</u> (of cells) corresponding to the measuring line on which they were analysed.
In fact, as the samples of a set X are, apart from special cases, analysed on measuring line X, the cells to be filled in in most cases are those surrounded by full borders near the diagonal.

- Under no circumstances should rows or columns be added to or deleted from the spreadsheet and, in particular, the cells containing latex grade data should remain cells C5 to Q37, and those contaiing CV grade data should remain C43 to Q75.

- Once the sheet has been completed, store it on the diskette, keeping the name SARInputForm, and send the diskette to ANRA.

II) Receipt of diskettes by ANRA and preparation of the common table under Excel.

This stage consists in compiling the results from the laboratories in a single common Excel spreadsheet. During this stage, the diskettes from each lab are inserted in the computer one after the other. By convention, for all subsequent processing operations, each laboratory is identified by the rank of its diskette in the order in which they were inserted. The order, i.e. the correspondence between rank and laboratory should be kept confidential by ANRA.

- The first operation at this stage is to open the SARTogether Excel file in the SARWork directory on the ANRA computer.
This sheet contains a macro which automatically loads the diskettes. If Excel asks for instructions, answer "Activate macros".

- The macro is designed to load *one* diskette. It must therefore be re-run as many times as there are diskettes.

This macro simply carries out two operations automatically: it opens the SARInputForm sheet supposed to be in drive "A:", and copies-pastes the contents of the sheet to the active cell in the SARTogether sheet. Two rules must be kept in mind for satisfactory operation of this macro:

a) The active cell (the cell surrounded by a double line or a bold line, which is usually the one just clicked on, whose contents are to be modified) is defined automatically each time. **It must not be another cell**. Take care therefore not to click just anywhere. If however, this does happen, the cells that should be active are:

Before the diskette is loaded	Active cell
1	E2
2	E68
3	E134
4	E200
5	E266

b) The macro does not function and an error message is displayed if there is no diskette in the drive when it is opened.
If there is a diskette in the drive, the macro does not know to which laboratory it corresponds, and does not check whether the data on the diskette have already been loaded.

Consequently, it is necessary to respect the following sequence of operations for each diskette:

. **Insert the diskette** in the drive,

. **Run the macro** "ChargeDisquetteLabo". Do this by:

+ Clicking Tools/Macros/Macros or Alt-F8.
+ Choose macro "ChargeDisquetteLabo"
+ Press the Run button

Or:

+ The macro can also be run directly via the keyboard short-cut "Ctrl+L".

. **Remove the diskette** from the drive once the macro run is finished, i.e. once you have control again and the active cell is the first cell of the next laboratory.

If a mistake is made, it is preferable to repeat the operation from the beginning.

III) Statistics calculations and back-up

Once all the diskettes have been loaded, the statistical analysis can be launched. For this operation, another macro called "Stats" is provided in the SARTogether.xls sheet. It can be run either from the "Tools/Macros/macros" menu or via the keyboard short-cut "Ctrl+s".

The macro first requests the number of the set of proficiency tests being analysed. Enter the number and click OK.

The macro then carries out all the desired calculations and plots the Youden graphs.

It creates as many worksheet tabs as there are laboratories, plus a worksheet tab entitled "Work", which is used for the calculations. Each worksheet tab contains the results of one laboratory.

Once the run is complete (which takes around two minutes), merely print out the results for each laboratory via the "File/Print" menu and send them off.

It is then necessary to make a back-up of the common table obtained, for the records. This is done as follows:

. Choose File/Save as...

. Save the sheet to the SARWork directory, under the name SARData*NNN*.xls, where *NNN* is the number of the curent SAR test cycle. The original folder "SARTogether.xls" must never be saved.

It should be noted that the "Common" worksheet tab can be saved in "Text (tabulation separator)" format for importing into the JMP software.

In the latter software, choose "File/Import" to read the file.
This can be useful particularly when carrying out synthesis analyses involving several sets of tests.

IV) Interpretation of results

- The "Stats" macro calculates the means of the 5 replications of each measurement for each instrument and each grade. The results obtained for all the participants are sorted in increasing order and listed to enable each laboratory to position itself in relation to the others.

The mean for the set of participants is calculated, along with the standard deviation between participants. As these two values are highly dependent upon the extreme values, it is the median of the set of results that is taken as the reference value. It appears from the results of the first set of tests that this convention is practical, objective and gives perfectly probable results.

- The difference between the value obtained by a laboratory and the reference value is judged from a scale defined after the first set of tests. Values called target standard deviations have been fixed for each property. These values are such that:

- If the difference between the lab value and the reference value is between 2 and 3 times the target standard deviation, the lab value is judged doubtful,

- If the difference is greater than 3 times the target standard deviation, the value is judged unacceptable.

The differences between lab and reference values are calculated and divided by the target standard deviation for each property and each lab. These "difference from target standard deviation" ratios are called Z-scores and must therefore be located within the 2 and 3 limits. An unacceptable Z-score indicates a probable calibration defect for the instrument in question.

- In order to assess the repeatability of measurements within each lab, the standard deviation between 5 replications of the same measurements is calculated. The mean of these standard deviations for all the participants is used as standard repeatability. The standard deviation of each participant is judged from the standard repeatability yardstick, by calculating the ratio of one with respect to the other. This "participant standard deviation from standard repeatability" ratio is called the K-score. If it is more than 2, it means that the instrument in question is two times less precise than standard repeatability, which is doubtful. If the K-score is over 3, the values obtained for the 5 replications need to be examined individually, to check whether an error has occurred at one moment or another.

- The Youden plot presenting the values obtained for one grade depending on the other, needs to be interpreted by its overall shape. If it is particularly elongated, it means that the values located at the extremities are affected by errors, which occurred substantially, twice in the same direction. This undoubtedly indicates a calibration defect for the instruments in question.
The confidence curve is centred on the mean of the values, and its radius is calculated from the interlaboratory standard deviation. Given the relatively small number of participants, and the sensitivity of these statistics to extreme values, this graph is not a particularly powerful tool for detecting doubtful values.